(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
17 May 2001 (17.05.2001)

PCT

(10) International Publication Number
WO 01/34871 A1

(51) International Patent Classification⁷: C23C 16/00, 16/08

(21) International Application Number: PCT/US00/30649

(22) International Filing Date: 6 November 2000 (06.11.2000)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
09/439,210 12 November 1999 (12.11.1999) US

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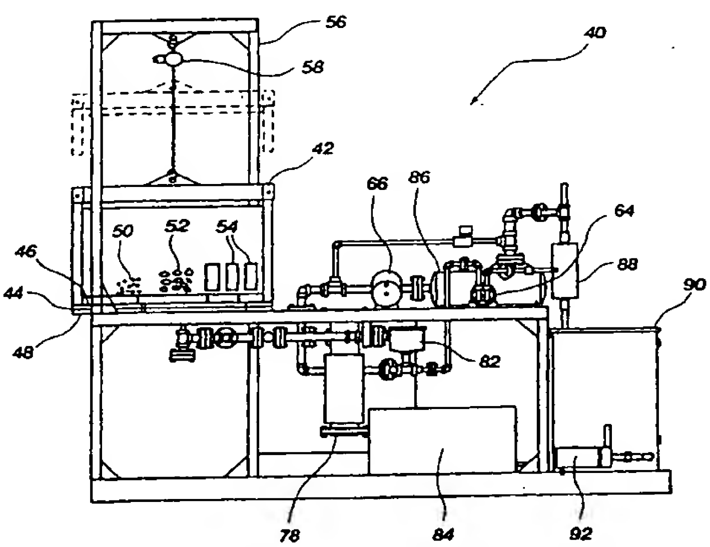
(81) Designated States *(national)*: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

(84) Designated States *(regional)*: ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published:
— *With international search report.*
— *Before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments.*

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: APPARATUS AND METHOD FOR PERFORMING SIMPLE CHEMICAL VAPOR DEPOSITION



(57) Abstract: An apparatus (40) for applying a protective metal coating to a substrate (54) surface by chemical vapor deposition. A reactor chamber (42) is provided in which the only opening exposable to atmosphere is at an open end having an annular flange (46) extending radially outward from the open end. A base plate (48) covers the opening, and a resilient ring (104) resides within an annular recess (106) formed in the plate (48) so that the ring is sandwiched between the base plate (48) and the flange (46) to provide a strong pressure-bearing seal. The seal is liquid cooled to prevent the ring from melting or oxygen from returning into the purged chamber, and the seal prevents harmful metallic vapor from leaking from the reactor chamber into the atmosphere. A local, liquid-to-gas argon supply (70) is used for producing cleaner, more oxygen-free argon. A cold trap (78) is used to remove harmful metallic salts from the exhaust.

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APPARATUS AND METHOD FOR PERFORMING SIMPLE CHEMICAL VAPOR DEPOSITION

BACKGROUND OF THE INVENTION

1. The Field of the Invention.

The present invention relates generally to chemical vapor deposition (CVD), and more particularly to a CVD apparatus for generating metallic vapor within a closed CVD reactor chamber itself.

2. Description of Related Art.

The acronym "CVD" refers to chemical vapor deposition. It is well known to provide a protective metal coating by CVD processes. CVD processes have many industrial applications, including preparing electronic chips or miniaturized circuits on a small piece of silicon.

In a CVD process, a mixture of a gaseous, metallic vapor is produced. The metallic vapor is then placed into contact with a substrate article, all within a reactor chamber under high temperature. Through various chemical reactions known to those skilled in the CVD field, metal from the vapor becomes deposited onto the substrate part. The gaseous remainder may be transported back to a donor source for replenishment of the metallic content or discharged from the reactor chamber.

Those skilled in the CVD field will understand the distinction between "dynamic CVD" and "simple CVD." In a dynamic CVD process, the gaseous, metallic vapor is generated at some remote location away from the CVD reactor chamber. For example,

HCl gas can be passed through aluminum pellets to produce $AlCl_3$. Such pre-reacted metallic vapor is then transported to the CVD reactor chamber in which the substrate parts reside, and injected therein to contact, react with, and coat the substrate parts. As a part of the process, oxygen is purged from the CVD reactor chamber by injecting argon into the chamber, as oxygen interferes with the CVD reaction.

In significant contrast, in a simple CVD process the gaseous, metallic vapors are generated inside the CVD reactor chamber itself, where the substrate parts to be coated reside, instead of generating the metallic vapor off-site and thereafter transporting it to the CVD reactor chamber. This is accomplished by placing an activator powder such as ammonium chloride within the CVD reactor chamber, along with metallic donor material. When heated, the activator sublimates into a gas and reacts with the metallic donor material to produce the metallic vapor. As the vapor moves around inside the argon-filled chamber, it contacts the substrate parts to cause the CVD reaction. Argon is typically used as a means of excluding oxygen from the reaction.

The simple CVD apparatus and methods presently known in the art are characterized by several disadvantages that are addressed by the present invention. For example, such apparatus often produce inferior metallic coating for any of several reasons, such as by failing to provide an optimal purge of the oxygen from the reactor chamber, or by failing to provide sufficiently clean

metallic donor material for use in the reaction. Such apparatus sometimes also fail to remove harmful metallic content from the exhaust gas prior to discharging it into the atmosphere. Still further, the prior art apparatus may utilize a weak sand-seal that permits both harmful metallic vapor into the atmosphere during the CVD reaction itself or oxygen to re-enter the furnace during the process. The present invention minimizes, and in some aspects eliminates, the failures described above and other problems, by utilizing the methods and structural features described herein.

BRIEF SUMMARY AND OBJECTS OF THE INVENTION

It is therefore an object of the present invention to provide a CVD apparatus that has a stronger seal on the reactor chamber.

It is another object of the present invention to provide such a CVD apparatus that is capable of removing oxygen to lower levels within the reactor chamber.

It is a further object of the present invention to provide a CVD apparatus that produces higher quality protective coatings.

The above objects and others not specifically recited are realized in a specific illustrative embodiment of an apparatus for applying a protective metal coating to a substrate surface by chemical vapor deposition. A reactor chamber is provided in which the only opening exposable to atmosphere is at an open end having an annular flange extending radially outward from the open end. A base plate is held against the flange such that the base plate covers the opening. A resilient o-ring resides within an annular recess formed in the plate so that the ring is sandwiched between the base plate and the flange to provide a strong pressure-bearing seal. This seal is liquid cooled to prevent the o-ring from melting or oxygen from returning into a purged chamber. The seal prevents harmful metallic vapor from leaking from the reactor chamber into the atmosphere. Oxygen is purged from the reactor chamber by a series of repeated argon pulses in alternating tandem with repeated applications of subatmospheric pressure to the interior of the reactor chamber. A local,

liquid-to-gas argon supply is used for producing cleaner, more oxygen-free argon to use in the oxygen purge. A cold trap is applied to the exhaust gas stream for removing environmentally unfriendly metallic salts from the exhaust before any vapor is discharged into the atmosphere.

In the current embodiment a small furnace with a round shape was constructed for the purpose of providing shorter cycle times in a manner of the Japanese KAIZAN manufacturing technologies.

Additional objects and advantages of the invention will be set forth in the description which follows, and in part will be apparent from the description, or may be learned by the practice of the invention without undue experimentation. The objects and advantages of the invention may be realized and obtained by means of the instruments and combinations particularly pointed out in the appended claims.

BRIEF DESCRIPTION OF THE DRAWINGS

The above and other objects, features and advantages of the invention will become apparent from a consideration of the subsequent detailed description presented in connection with the accompanying drawings in which:

FIG. 1 is a schematic view of a prior art simple CVD apparatus utilizing a sand seal;

FIG. 2 is a side view of a simple CVD apparatus, made in accordance with the principles of the present invention;

FIG. 3 is a plan view of the simple CVD apparatus of FIG. 2;

FIG. 4 is an exploded view of a reactor chamber of the simple CVD apparatus of FIGS. 2 and 3;

FIG. 4A is a break-away, enlarged view of a base plate portion of the reactor chamber of FIG. 4;

FIG. 4B is a break-away, enlarged view of a different base plate portion of the reactor chamber of FIG. 4;

FIG. 5 is a view of an upper surface of the base plate of the reactor chamber of FIG. 4;

FIG. 6 is a view of an under surface of the base plate of FIG. 5;

FIG. 7 is a side view of an assembled version of the reactor chamber of FIG. 4; and

FIG. 8 is a side, cross-sectional view the assembled version of the reactor chamber of FIG. 7.

DETAILED DESCRIPTION OF THE INVENTION

For the purposes of promoting an understanding of the principles in accordance with the invention, reference will now be made to the embodiments illustrated in the drawings and specific language will be used to describe the same. It will nevertheless be understood that no limitation of the scope of the invention is thereby intended. Any alterations and further modifications of the inventive features illustrated herein, and any additional applications of the principles of the invention as illustrated herein, which would normally occur to one skilled in the relevant art and having possession of this disclosure, are to be considered within the scope of the invention claimed.

Applicant has discovered that protective metallic coatings produced by chemical vapor deposition (referred to herein as "CVD") can be made with higher quality and conducted in a more environmentally safe manner by utilizing the structural features and procedures described below. To whatever extent the following references may be useful as background or as disclosure in aid to the present disclosure, said following references are incorporated by reference herein in their entireties: U.S. Patent No. 4,427,720 (issued January 24, 1984 to Gauje) and French Patent No. 1,433,497.

Briefly stated, the principles of the present invention include utilizing a CVD reactor chamber with a water-cooled O-ring seal to produce a stronger, pressure-bearing seal that prevents oxygen from entering the chamber and prevents metallic

vapor from seeping out of the chamber into the atmosphere. A cleaner argon supply is used for injecting argon into the reactor chamber prior to, and during, the CVD reaction to purge oxygen from the chamber. The argon supply is local and utilizes a much shorter passage between the supply and the reactor chamber and with fewer connections, to minimize oxygen seepage into the argon movement path. A liquid-ring vacuum pump is used for withdrawing oxygen from the reactor chamber in alternating tandem with argon purge pulses into the chamber. Cleaner metallic donor material (having less than 20 ppm of sulfur as a tramp contaminant) is used in the CVD reaction. Unreacted vapors remaining at the end of the cycle are removed from the waste exhaust to reduce to a negligible amount the contaminants being injected into the atmosphere, thereby keeping the environment safe.

Referring now to FIG. 1, there is shown a prior art version of a simple CVD device, designated generally at 10. The prior art CVD device 10 includes an inner chamber 12 disposed within an outer chamber 14, both residing in a sealing base 16. Argon gas is transported from a remote argon source 18, along a gas line 20, into the inner chamber 12 and outer chamber 14. The argon gas line 20 is typically at least 200 feet long, and often has numerous connection points 22 along its length. Activator powder 24 and metallic donor material 26 are placed within the inner chamber 12, along with one or more parts 28 to be coated. Sand 30 is placed within the sealing base 16 to seal the inner chamber 12 and outer chamber 14 from atmosphere. The outer

chamber 14 encloses the internal reactor vessel.

In operation, argon is transported through the line 20 to purge oxygen from the inner chamber 12 and outer chamber 14 at the start of a cycle, and is shut off when heating commences. The device 10 is heated to cause the activator powder 24, typically ammonium bi-fluoride, to sublimate into a gas which then reacts with the metallic donor material 26 to produce a gaseous, metallic vapor. The vapor then reacts with an exterior surface of the part 28 and thereby causes the metallic content of the vapor to become deposited into the surface of the part as a coating, by various chemical reactions known and understood by those skilled in the CVD field. After the coating reaction is complete, the remainder of the gaseous metallic vapor is discharged into the atmosphere.

The prior art CVD device 10 and corresponding process are characterized by several disadvantages. First of all, the sand seal 30 cannot withstand pressure. As a result it may leak harmful metallic vapor into the atmosphere and it cannot be used in conjunction with a vacuum pump. Further, as those skilled in the field of fluid mechanics will appreciate, the argon supply 18 is positioned so far away from the reactor chamber 12 that several leak points are possible along the gas line 20 at its many connection points 22. This results in oxygen being withdrawn from the atmosphere into the gas line 20 at any leak point that might exist along the line 20, because the rush of argon travel produces a subatmospheric pressure point at any leak

opening along the line 20 as those of ordinary skill will understand. The presence of oxygen within the reactor chamber 12 interferes with the CVD reaction and results in an inferior quality coating being deposited on the part 28.

Referring now to FIGS 2-3, there is shown a simple CVD apparatus designated generally at 40, made in accordance with the principles of the present invention. The apparatus 40 includes a reactor chamber 42, also called a retort can. The only passageway from the interior of the chamber 42 to the atmosphere is the path 44 residing between a flange 46 and a base plate 48. A resilient O-ring (not shown in FIGS. 2-3) resides sandwiched between the flange 46 and plate 48 to seal the interior of the chamber 42 from communication with atmosphere.

The apparatus 40 further includes a frame 56 and a lifting motor 58 for lifting the reactor chamber 42 as needed. An argon inlet port 60 is formed in a base of the reactor chamber 42. A discharge port 62 is also formed in a base of the reactor chamber 42, which is disposed in communication with a liquid ring vacuum pump 64 and also an exhaust blower 66. The vacuum pump 64 is selectively blocked from communication with the discharge port 62 by an actuator valve 68, and the exhaust blower is also valved in its communication with discharge port 62.

A local liquid-to-gas argon supply 70 is disposed in communication with the inlet port 60 by an argon movement path that passes through a short gas line 72, through a sealed control panel 74 and from the panel 74 to the inlet port 60. The concept

and operation of a "liquid-to-gas" argon supply is known to those of ordinary skill in the field as a cleaner source of argon (converting liquid argon to gas results in an ultra high purity), in part because there is less opportunity for contamination of the gas since the argon resides in liquid form until shortly before being deployed into the reactor chamber 42 through passages that have been sealed with solder (preferably silver solder) to prevent infiltration.

In operation, activator powder 50 and chunklets of metallic donor material 52 are placed inside the reactor chamber 42, along with substrate parts 54 to be coated. Argon gas is pumped from the argon supply 70 into the reactor chamber 42 via the inlet port 60, preferably as a series of pulses (although a continuous feed could also be used), and subatmospheric pressure is applied to the discharge port 62 by the vacuum pump 64 between the argon pulses in alternating tandem with the argon supply 70, by operation of the valving. In this manner, oxygen is alternately displaced from the reactor chamber 42 by the argon pulses, and further withdrawn by the applications of subatmospheric pressure between the argon pulses. The vacuum flow path passes through a filter 82, which removes harmful metallic content from the vacuum stream before it passes to the atmosphere. The vacuum pump 64 is preferably a liquid ring pump as known to those of ordinary skill in the field. In the present invention, a liquid ring vacuum pump preferably capable of producing subatmospheric pressure of less than 70 Torr is provided (although a higher

subatmospheric pressure is also workable), and more preferably the subatmospheric pressure is less than 60 Torr, and most preferably the subatmospheric pressure is 50 Torr. Alternatively, a pump utilizing a sealant liquid other than water, such as silicon-based oil, may be capable of producing a subatmospheric pressure as low as 20 Torr.

The reactor chamber 42 and its contents are heated to a high temperature, preferably in the range 1925 degrees Fahrenheit to 2000 degrees Fahrenheit, for example when producing a coating of NiAl (nickel aluminide), or lower or higher if producing other coatings. This causes the activator powder 50 to sublimate into a gas, which then reacts with the chunklets of metallic donor material 52 to produce a gaseous, metallic vapor. The metallic vapor then reacts with an exterior surface of the parts 54 and thereby causes the metallic content of the vapor to become deposited into the surface of the parts 54 as a protective metallic coating, by various chemical reactions known and understood by those skilled in the CVD field. Simultaneously, aluminum or other metal diffuses into the coated substrate.

After the coating reaction is complete, the remainder of the gaseous metallic vapor is withdrawn from the reactor chamber 42 through the discharge port 62 by the blower 66. The dashed line 76 represents the exhaust path. Before the remainder of the vapor passes to the atmosphere, it is exposed along its exhaust path 76 to cold trap 78 and a filter 80, by which the harmful metallic content is removed as a metallic salt from the exhaust

remainder, in a manner understood by those skilled in the art of cold traps and filters. This operates to reduce the amount of metallic vapor discharged into the atmosphere to a negligible, harmless amount, preferably less than 15 grams of harmful waste content per batch cycle, more preferably less than 10 grams per batch cycle, and most preferably less than 7 grams per batch cycle. The cold trap 78 is operated by a refrigeration unit 84 as known to those skilled in the field, and it is to be understood that the cold trap could be replaced by any other suitable contaminant removal device, such as a wet scrubber as known to those skilled in the field. A silencer 86 reduces noise produce by the exhaust and contaminant removal. The waste content of metallic salts removed from the metallic vapor used in the CVD reaction is removed from the exhaust path 76 and trapped in a separator 88.

During the high temperature CVD reaction, the base plate 48 is cooled with chilled water from tank 90, which is pumped to the plate 48 by water pump 92 as described below in more detail. This maintains the base plate 48 at a temperature of about 200 degrees Fahrenheit, thus enabling the plate 48 to be made from less expensive metal such as stainless steel.

Referring now to FIGS. 4, 4A and 4B, there is shown an exploded view of the reactor chamber 42. The reactor chamber 42 includes an upper cylinder 100 having a single, lower opening 102 and the flange 46 extending radially outwardly from said opening 102. The base plate 48 is placed against the flange 46 such that

a resilient O-ring 104, residing in an annular recess 106 formed in an upper surface of the base plate 48, becomes sandwiched between the flange 46 and base plate 48 to produce a strong, pressure bearing seal capable of withstanding at least 15 psi of pressure. The O-ring 104 is thus a one-piece, unitary resilient ring, and not a barrier composed of accumulated-but-unattached particles such as the sand seal 30 shown in FIG. 1. The weight of the chamber 42, which is preferably at least 325 pounds, bears down upon the O-ring 104 to produce a pressure-bearing seal. Bolts 51 (shown in FIG. 7) can be tightened to add more pressure.

A second, inner annular recess 108 is also provided, in which a fiberglass ring 110 is placed to aid in producing an even stronger seal. Annular protrusions 112 and 114 are formed on an under surface of the flange 46, and are respectively aligned with the outer annular recess 106 and the inner annular recess 108 to aid in more effectively and strongly sandwiching the sealing O-ring 104 and fiberglass ring 110 between the base plate 48 and the flange 46 under the weight of the reactor chamber 42. The rings 104 and 110 may be made from any suitable material capable of producing a seal, preferably a resilient material.

Referring now to FIG. 5, there is shown a second inlet port 112 formed in the base plate 48 between the outer annular recess 106 and the inner annular recess 108. The liquid-to-gas argon supply 70 is disposed in communication with this second inlet port 112 as well, for producing an argon flow between the sealing rings 104 and 110 to further ensure the blockage of oxygen from

entering into the reactor chamber 42. The base plate 48 may include internal reinforcing ribs 49 for increased strength.

Beneath the base plate 48, on an under surface thereof, is disposed a tubular coil 114, through which chilled water is pumped by the water pump 92. Also, insulation 122 is provided between the base plate 48 and the upper cylinder 100 as shown most clearly in FIG. 8. The cooling coil 114 and the insulation 122 enable the base plate to be made of less expensive metal, preferably stainless steel, such as 304 SS. The upper cylinder 100 is preferably made from a ferrous alloy containing nickel, such as RA333, RA330, Inconel600 or Inconel601, as those varieties are known to those of ordinary skill in the field of alloys. Such alloys enable the upper cylinder to withstand repeated heat applications of at least 2000 degrees Fahrenheit for sixteen hours or more without failure or excessive creep of the material.

Referring now again to FIG. 3, it is to be understood that the argon gas conveyed from the argon supply 70 (the argon gas being of an ultra high purity by being converted from liquid argon) to the inlet port 60 travels along a passageway defined by the gas line 72 that is less than twenty feet long, and more preferably less than fifteen feet long, and, if desired and feasible, less than ten feet long. This enables a shorter argon gas movement path with fewer connections 73a, 73b and therefore fewer leak opportunities for the argon flow to withdraw oxygen from the environment. Since the gas line 72 passes from the

supply 70 into the pressurized panel 74 and then directly to the inlet port 60, at least all except two connections along the argon gas line 72 passageway are selected from the group consisting of a soldered connection (preferably silver soldered) and a connection residing within the pressurized panel 74, which is a pressurized enclosure. Most preferably, the only connection that is not either a silver soldered connection or a connection residing within the pressurized panel 74 is connection 73b, and the risk of oxygen leakage into the argon flow path is thereby significantly reduced by the present invention.

As shown in FIG. 8, a heating means 120 is included for heating the reactor chamber 42, specifically the upper cylinder 100 and any contents residing therein, to a degree sufficient to cause the reactants 50 and 52 within said reactor chamber 42 to produce a metallic vapor which thereafter reacts with a surface of the substrate parts 54 to deposit a protective metal coating on said surface. The heating means 120 is preferably a heater element ring made from a suitable ceramic, in which a series of metal windings (not shown in FIG. 8) are embedded. The metal windings are preferably made from Kanthal, which is known chemically as FeCrAl, and an AC voltage is applied to the windings to cause the wires to heat. The design of a ceramic heating ring in which Kanthal wire coils are embedded is for the purpose of producing a longer life of the heating means 120.

The heating means comprises a means for raising the temperature of the reactants 50 and 52 within the reactor chamber

42 to within a range of approximately 1900 degrees fahrenheit to 2100 degrees fahrenheit, and more preferably to within a range of approximately 1925 degrees fahrenheit to 2000 degrees fahrenheit.

The chunklets of metallic donor material 52 are preferably selected from the group consisting of chromium, aluminum, and mixtures of chromium and aluminum. Chromium is preferably added to control the melting point of aluminum and otherwise plays only a minor role in the chemical reaction. The chunklets of metallic donor material 52 are therefore preferably a mixture of chromium and aluminum, comprising any suitable stoichiometric mixture within a range of 1:99 to 99:1 Cr-Al. The mixture is preferably either 56/44 Cr-Al or 70/30 Cr-Al, but it could also be 42/58 Cr-Al, 50/50 Cr-Al, or any other suitable stoichiometric mixture. The chunklets 52 preferably comprise less than 6.0 parts per million of sulfur, and more preferably even less.

In accordance with the features and combinations described above, a preferred process for applying a protective metal coating to a substrate surface by chemical vapor phase deposition, comprises the steps of:

(a) selecting a reactor chamber comprising a cylinder having an opening and a flange extending radially outwardly from said opening, and a base plate removably disposed against the flange such that said base plate removably covers the opening;

(b) placing into the reactor chamber at least one substrate part and reactants capable of reacting to produce a metallic

vapor;

(c) injecting argon gas into the interior of the reactor chamber to thereby purge oxygen from said reactor chamber by conveying argon gas from an argon source into the reactor chamber along a movement path between said argon source and reactor chamber that is less than twenty feet long;

(d) heating the reactor chamber and its contents to a degree sufficient to cause the reactants within said reactor chamber to produce a metallic vapor, for a duration of time sufficient to cause said metallic vapor to move into contact with the at least one substrate part such that said metallic vapor reacts with a surface of the substrate part to deposit a protective metal coating on said surface;

(e) reducing or eliminating the heat applied to the reactor chamber to permit said reactor chamber to cool in accordance with ambient temperature; and

(f) removing the contents of the reactor chamber.

It is to be understood that the above-described arrangements are only illustrative of the application of the principles of the present invention. Numerous modifications and alternative arrangements may be devised by those skilled in the art without departing from the spirit and scope of the present invention and the appended claims are intended to cover such modifications and arrangements. Thus, while the present invention has been shown in the drawings and fully described above with particularity and

detail in connection with what is presently deemed to be the most practical and preferred embodiment(s) of the invention, it will be apparent to those of ordinary skill in the art that numerous modifications, including, but not limited to, variations in size, materials, shape, form, function and manner of operation, assembly and use may be made without departing from the principles and concepts set forth herein.

CLAIMS

What is claimed is:

1. An apparatus for applying a protective metal coating to a substrate surface by simple chemical vapor phase deposition, said apparatus comprising:

a reactor chamber for holding therein at least one substrate part and reactants capable of reacting to produce a metallic vapor;

heating means for heating the reactor chamber and any contents residing therein, to a degree sufficient to cause reactants within said reactor chamber to produce a metallic vapor which thereafter reacts with a surface of the substrate part to deposit a protective metal coating on said surface; and

sealing means for sealing the reactor chamber to a degree sufficient to prevent oxygen passage into said reactor chamber, said sealing means comprising at least one one-piece, unitary resilient member disposed within an annular passage of said reactor chamber.

2. The apparatus of claim 1, wherein the sealing means is characterized by an absence of a barrier composed of accumulated-but-unattached particles.

3. The apparatus of claim 1, wherein the resilient ring is disposed between a flange of the reactor chamber and a base plate removably disposed against said flange.

4. The apparatus of claim 1, wherein the sealing means further comprises a means for producing a pressure bearing seal capable of withstanding at least ten psi without failure of the seal.

5. The apparatus of claim 4, wherein the sealing means further comprises a means for producing a pressure bearing seal capable of withstanding at least fifteen psi without failure of the seal.

6. The apparatus of claim 1, further comprising a purging means for purging oxygen from the reactor chamber.

7. The apparatus of claim 6, wherein the purging means further comprises means for reducing oxygen content within the reactor chamber to less than two parts per million.

8. The apparatus of claim 6, wherein the purging means further comprises subatmospheric pressure means for applying subatmospheric pressure to an interior of the reactor chamber.

9. The apparatus of claim 8, wherein the subatmospheric pressure means further comprises means for applying a subatmospheric pressure of less than 70 Torr to the interior of the reactor chamber.

10. The apparatus of claim 8, wherein the subatmospheric pressure means further comprises means for applying a subatmospheric pressure of less than 45 Torr to the interior of the reactor chamber.

11. The apparatus of claim 8, wherein the subatmospheric pressure means further comprises means for applying a subatmospheric pressure of less than 20 Torr to the interior of the reactor chamber.

12. The apparatus of claim 6, wherein the purging means comprises a local gas source, and a passageway extending from said gas source into communication with the reactor chamber for conveying gas from said gas source into said reactor chamber, wherein said passageway defines a gas movement path from the gas source to the reactor chamber that is less than twenty feet long.

13. The apparatus of claim 12, wherein the gas movement path defined by the passageway is less than fifteen feet long.

14. The apparatus of claim 12, wherein the gas movement path defined by the passageway is less than ten feet long.

15. The apparatus of claim 12, wherein at least all except two connections along the passageway are selected from the group consisting of:

a soldered connection; and

a connection residing within a pressurized enclosure.

16. The apparatus of claim 15, wherein at least all except one connection along the passageway is selected from the group consisting of:

a soldered connection; and

a connection residing within a pressurized enclosure.

17. The apparatus of claim 6, wherein the purging means comprises a liquid-to-gas argon supply means disposed in communication with an interior of the reactor chamber for injecting argon gas into said reactor chamber and thereby forcing oxygen from said reactor chamber.

18. The apparatus of claim 17, wherein the purging means further comprises a valve means for selectively blocking and releasing argon flow into the reactor chamber.

19. The apparatus of claim 17, wherein the liquid-to-gas argon supply means further comprises means for alternately blocking and releasing argon gas flow into the reactor chamber to thereby inject the argon gas into said reactor chamber as a series of pulses.

20. The apparatus of claim 19, wherein the purging means

further comprises subatmospheric pressure means, operating in alternating tandem with the liquid-to-gas argon supply means, for applying a pulse of subatmospheric pressure to an interior of the reactor chamber between pulses of argon gas injections.

21. The apparatus of claim 20, wherein the subatmospheric pressure means comprises a liquid ring vacuum pump.

22. The apparatus of claim 20, wherein the subatmospheric pressure means includes means for applying a subatmospheric pressure of less than 70 Torr to the interior of the reactor chamber.

23. The apparatus of claim 1, wherein the heating means comprises means for raising the temperature of the reactants within the reactor chamber to at least 1500 degrees fahrenheit.

24. The apparatus of claim 23, wherein the heating means comprises means for raising the temperature of the reactants within the reactor chamber to within a range of approximately 1900 degrees fahrenheit to 2100 degrees fahrenheit.

25. The apparatus of claim 23, wherein the heating means comprises means for raising the temperature of the reactants within the reactor chamber to within a range of approximately 1925 degrees fahrenheit to 2000 degrees fahrenheit.

26. The apparatus of claim 1, further comprising an exhaust means for selectively exhausting the metallic vapor from the reactor chamber as an exhaust gas stream.

27. The apparatus of claim 26, further comprising a removal means for removing metallic salts from the waste gas stream.

28. The apparatus of claim 27, wherein the removal means comprises a cold trap.

29. The apparatus of claim 28, wherein the exhaust means includes a hollow passage disposed in communication with an interior of the reactor chamber, and wherein the cold trap is disposed in communication with the hollow passage.

30. The apparatus of claim 27, wherein the removal means comprises a wet scrubber.

31. The apparatus of claim 1, further comprising reactants capable of reacting to produce a metallic vapor, said reactants being selected from the group consisting of chromium, aluminum, mixtures of chromium and aluminum, and ammonium bi-fluoride.

32. The apparatus of claim 31, wherein the mixture of chromium and aluminum comprises a stoichiometric mixture within a range of 1:99 to 99:1 Cr-Al.

33. The apparatus of claim 32, wherein the mixture of chromium and aluminum comprises a stoichiometric mixture of 50/50 Cr-Al.

34. The apparatus of claim 32, wherein the mixture of chromium and aluminum comprises a stoichiometric mixture of 42/58 Cr-Al.

35. The apparatus of claim 32, wherein the mixture of chromium and aluminum comprises a stoichiometric mixture of 56/44 Cr-Al.

36. The apparatus of claim 32, wherein the mixture of chromium and aluminum comprises a stoichiometric mixture of 70/30 Cr-Al.

37. The apparatus of claim 1, further comprising reactants capable of reacting to produce a metallic vapor, said reactants comprising less than 6.0 parts per million of sulfur.

38. The apparatus of claim 1, wherein the reactor chamber comprises a retort chamber made from a ferrous alloy containing nickel.

39. The apparatus of claim 38, wherein the ferrous alloy containing nickel is selected from the group consisting of RA333,

RA330, Inconel600 and Inconel601.

40. The apparatus of claim 1, wherein the reactor chamber comprises a cylinder having an opening and a flange extending radially outwardly from said opening, and a base plate removably disposed against the flange such that said base plate removably covers the opening.

41. The apparatus of claim 40, wherein the base plate is made from stainless steel.

42. The apparatus of claim 41, wherein the base plate is made from a variety of stainless steel known as 304 SS.

43. The apparatus of claim 40, further comprising cooling means disposed on an external surface of the base plate for cooling the base plate.

44. The apparatus of claim 43, wherein the cooling means further comprises a tubular member disposed on the external surface of the base plate, and a means for passing a coolant through the tubular member.

45. The apparatus of claim 44, wherein the coolant comprises water.

46. The apparatus of claim 1, wherein the sealing means further comprises a second annular passage of the reactor chamber in which a second one-piece, unitary resilient ring resides.

47. The apparatus of claim 46, wherein the sealing means further comprises an injection port disposed between the first and second annular passages and a means for injecting argon gas between said first and second annular passages.

48. A process for applying a protective metal coating to a substrate surface by chemical vapor phase deposition, said method comprising the steps of:

(a) selecting a reactor chamber comprising a cylinder having an opening and a flange extending radially outwardly from said opening, and a base plate removably disposed against the flange such that said base plate removably covers the opening;

(b) placing into the reactor chamber at least one substrate part and reactants capable of reacting to produce a metallic vapor;

(c) injecting argon gas into the interior of the reactor chamber to thereby purge oxygen from said reactor chamber by conveying argon gas from an argon source into the reactor chamber along a movement path between said argon source and reactor chamber that is less than twenty feet long; and

(d) heating the reactor chamber and its contents to a degree sufficient to cause the reactants within said reactor chamber to

produce a metallic vapor, for a duration of time sufficient to cause said metallic vapor to move into contact with the at least one substrate part such that said metallic vapor reacts with a surface of the substrate part to deposit a protective metal coating on said surface.

49. The process of claim 48, wherein step (c) further comprises conveying the argon gas along a movement path between the argon source of the reactor chamber that is less than fifteen feet long.

50. The process of claim 48, wherein step (a) further comprises placing a resilient ring between the flange of the reactor chamber and the base plate removably disposed against said flange, to thereby seal the reactor chamber to a degree sufficient to prevent oxygen passage into said reactor chamber, and forcing the base plate against the flange with enough force sufficient to produce a seal that can withstand at least 10 psi of pressure.

51. The process of claim 48, wherein step (c) further comprises conveying argon along a movement path of less than twenty feet that is defined by a passageway along which at least all except two connections along said passageway are selected from the group consisting of:

a soldered connection; and

a connection residing within a pressurized enclosure.

52. An apparatus for applying a protective metal coating to a substrate surface by chemical vapor phase deposition, said apparatus comprising:

a reactor chamber for holding therein at least one substrate part and reactants capable of reacting to produce a metallic vapor;

heating means for heating the reactor chamber and any contents residing therein, to a degree sufficient to cause reactants within said reactor chamber to produce a metallic vapor which thereafter reacts with a surface of the substrate part to deposit a protective metal coating on said surface;

sealing means for sealing the reactor chamber to a degree sufficient to prevent oxygen passage into said reactor chamber, said sealing means comprising at least a one-piece, unitary resilient ring disposed within an annular passage of said reactor chamber; and

purging means for purging oxygen from the reactor chamber, said purging means comprising:

a liquid-to-gas argon supply means disposed in communication with an interior of the reactor chamber for injecting argon gas into said reactor chamber and thereby forcing oxygen from said reactor chamber; and

subatmospheric pressure means for applying subatmospheric pressure to an interior of the reactor

chamber.

53. The apparatus of claim 52, wherein the purging means comprises a local gas source, and a passageway extending from said gas source into communication with the reactor chamber for conveying gas from said gas source into said reactor chamber, wherein said passageway defines a gas movement path that is less than twenty feet long, the apparatus further comprising:

an exhaust means for selectively exhausting the metallic vapor from the reactor chamber as an exhaust gas stream;

a removal means for removing metallic salts from the waste gas stream; and

reactants capable of reacting to produce a metallic vapor, said reactants comprising less than 6.0 parts per million of sulfur.

54. The apparatus of claim 53, wherein the removal means comprises a cold trap.

55. The apparatus of claim 53, wherein the removal means comprises a wet scrubber.

56. The apparatus of claim 53:

wherein the reactants further comprise reactants capable of reacting to produce a metallic vapor, said reactants being selected from the group consisting of chromium, aluminum,

mixtures of chromium and aluminum, and ammonium bi-fluoride;

wherein the sealing means is characterized by an absence of a barrier composed of accumulated-but-unattached particles;

wherein the resilient ring is disposed between a flange of the reactor chamber and a base plate removably disposed against said flange;

wherein the sealing means further comprises a means for producing a pressure bearing seal capable of withstanding at least ten psi without failure of the seal;

wherein the purging means further comprises means for reducing oxygen content within the reactor chamber to less than two parts per million;

wherein the subatmospheric pressure means further comprises means for applying subatmospheric pressure to an interior of the reactor chamber of less than 70 Torr;

wherein at least all except two connections along the passageway of the purging means are selected from the group consisting of:

a soldered connection; and

a connection residing within a pressurized enclosure;

wherein the liquid-to-gas argon supply means further comprises means for alternately blocking and releasing argon gas flow into the reactor chamber to thereby inject the argon gas into said reactor chamber as a series of pulses;

wherein the heating means comprises means for raising the temperature of the reactants within the reactor chamber to at

least 1500 degrees fahrenheit;

wherein the mixture of chromium and aluminum comprises a stoichiometric mixture within a range of 1:99 to 99:1 Cr-Al;

wherein the reactor chamber comprises a cylinder having an opening and a flange extending radially outwardly from said opening, and a base plate removably disposed against the flange such that said base plate removably covers the opening;

wherein the apparatus further comprises cooling means disposed on an external surface of the base plate for cooling the base plate.

57. The apparatus of claim 53, wherein the subatmospheric pressure means further comprises a means, operating in alternating tandem with the liquid-to-gas argon supply means, for applying a pulse of subatmospheric pressure to an interior of the reactor chamber between pulses of argon gas injections.

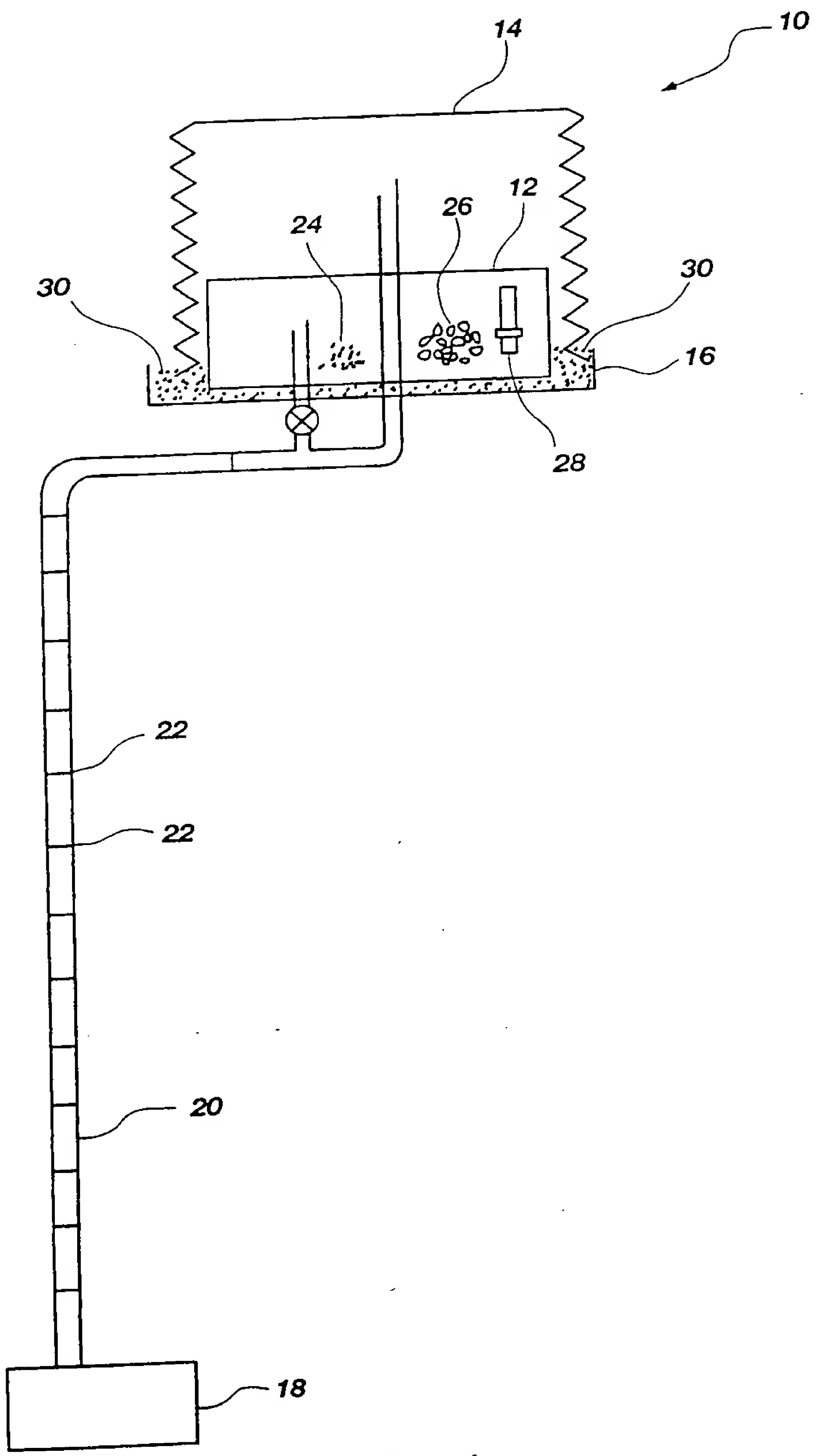

Fig. 1
(PRIOR ART)

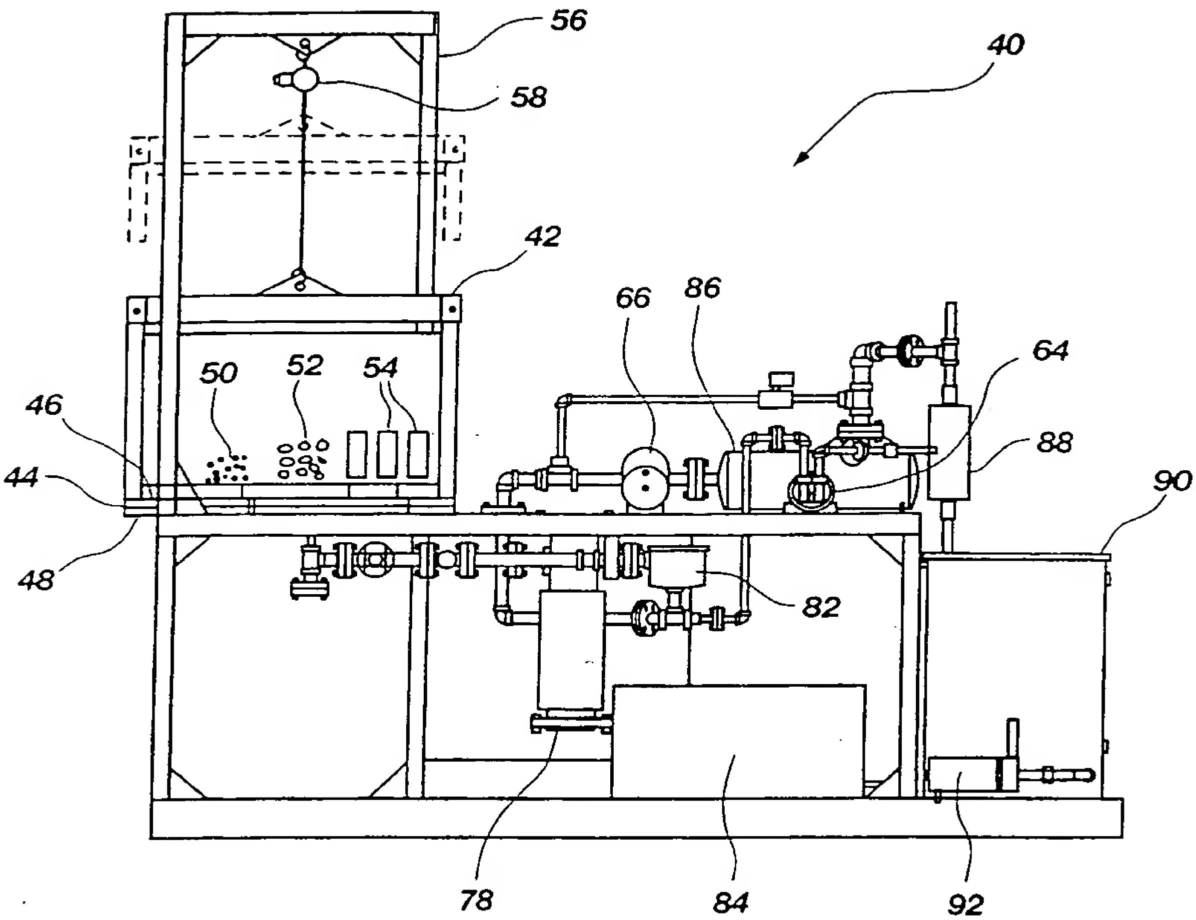
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Fig. 2

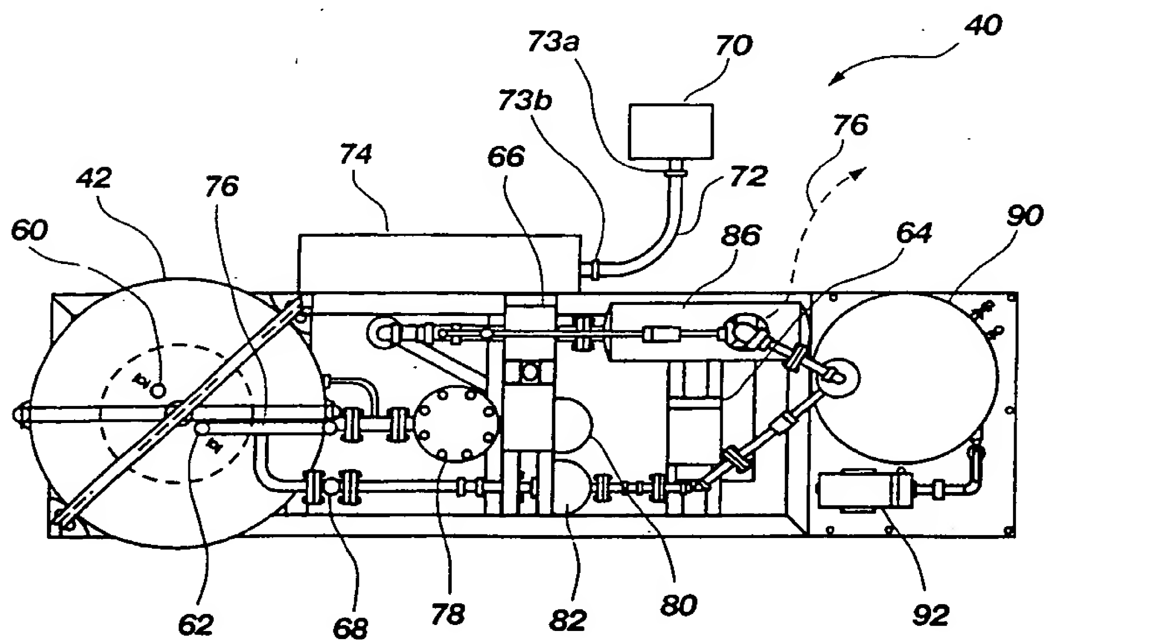
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Fig. 3

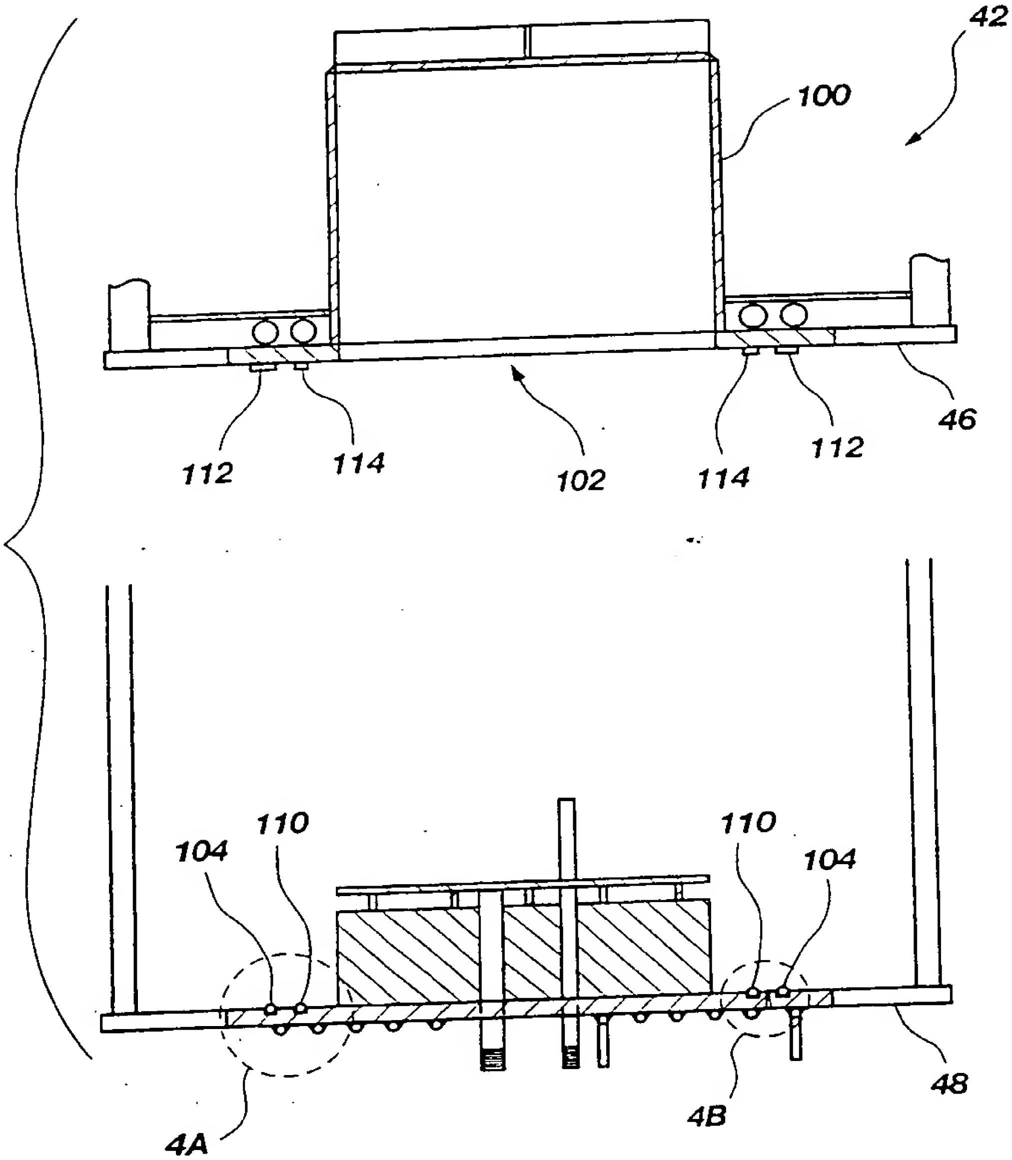

Fig. 4

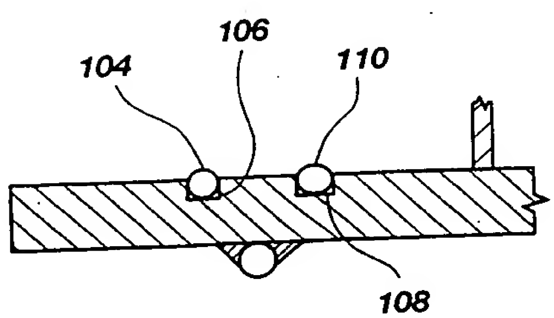

Fig. 4A

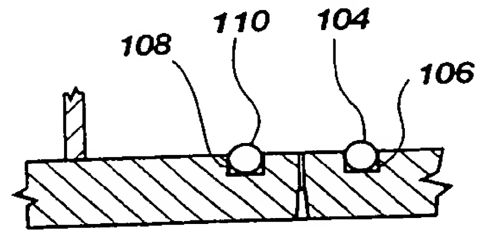

Fig. 4B

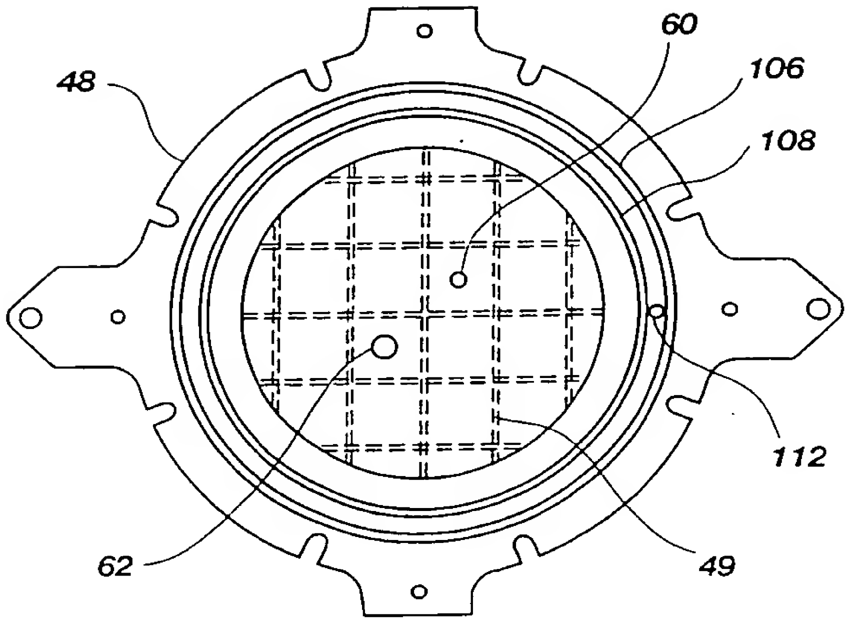

Fig. 5

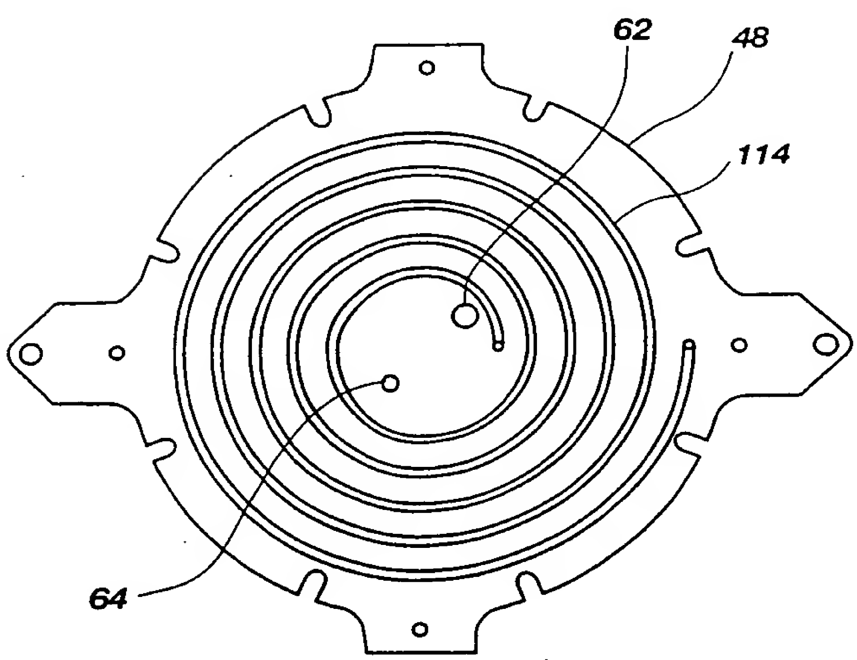

Fig. 6

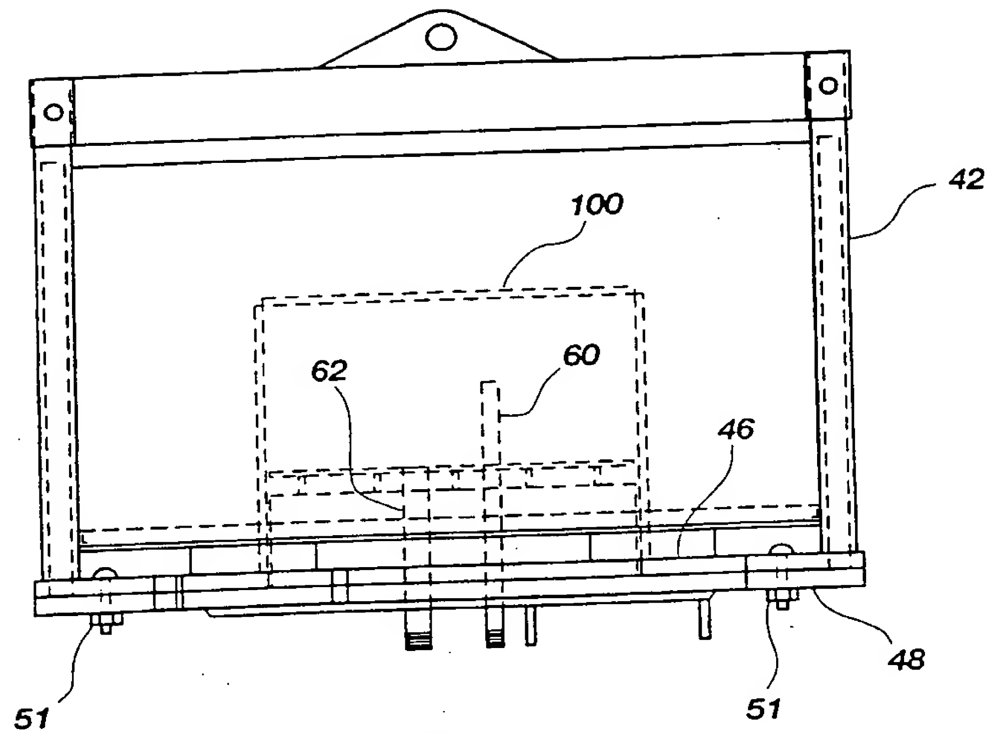

Fig. 7

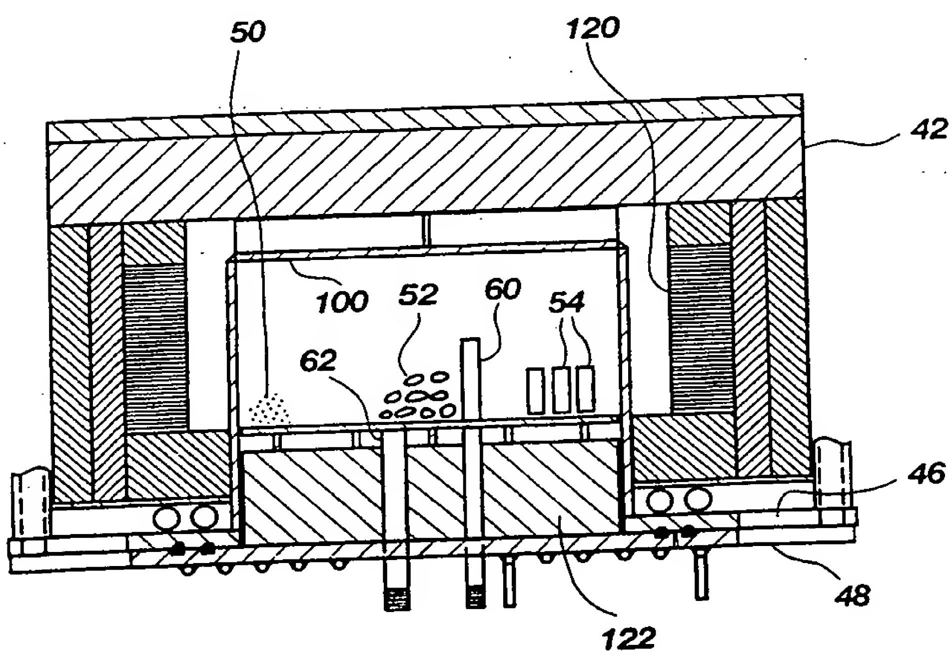

Fig. 8

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US00/30649

A. CLASSIFICATION OF SUBJECT MATTER
IPC(7) :C23C 16/00, 16/08.
US CL :118/733, 723VE; 427/248.1, 252, 253.
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 118/733, 723VE, 724, 725, 717; 427/248.1, 252, 253.

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

Please See Extra Sheet.

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X --- Y	US 3,329,601 A (MATTOX) 04 July 1967, entire document.	1-11,23-26,31-37,40 ---------- 12-22, 27-30, 38, 39, 41-57
X --- Y	US 3,507,248 A (SEELEY et al) 21 April 1970, entire document.	1-11, 23-26, 31-37,40 ---------- 12-22, 27-30, 38, 39, 41-57
Y	US 4,396,213 A (HAWKINS) 02 August 1983, entire document.	15, 16

[X] Further documents are listed in the continuation of Box C. [ ] See patent family annex.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
12 MARCH 2001	04 APR 2001

Name and mailing address of the ISA/US	Authorized officer
Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231	JEFFRIE R. LUND
Facsimile No. (703) 305-3230	Telephone No. (703) 308-0661

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 5,261,963 A (BASTA et al) 16 November 1993, entire document.	1-57
Y	US 5,632,820 A (TANIYAMA et al) 27 May 1997, figure 2.	46, 47
Y	US 5,750,436 A (YAMAGA et al) 12 May 1998, figure 6 and 13.	6-11, 17-20, 22, 48-57
Y	US 5,820,641 A (GU et al) 13 October 1998, abstract.	27-29, 53, 54, 56, 57
Y	US 5,851,293 A (LANE et al) 22 December 1998, entire document.	30, 55
Y, P	US 6,083,321 A (LEI et al) 04 July 2000, entire document.	12-14, 17-20, 22, 48-57
Y,E	US 6,143,361 A (NEAR et al) 07 November 2000, entire document.	1-57

B. FIELDS SEARCHED

Electronic data bases consulted (Name of data base and where practicable terms used):

USPAT, EPO, JPO, DERWENT, IBM TDB
search terms: heat$3, seal$3, purge, chemical vapor deposition, CVD, sublimat$3, evaporat$3,c chromium, aluminum, ammonium chloride, argon, gasbomb, solder, cold trap.